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(54) Title: A BONDED FOAM AND A PROCESS FOR THE PRODUCTION THEREOF

(57) Abstract: The bonded foam comprises a plurality of isocyanate-based polymeric foam particles, in particular polyurethane foam particles, bonded to one another by means of an isocyanate based binder. In order to make this foam suitable for applications, in particular for floor application, it further comprises at least one water-repellent additive and has a water uptake, determined at 25 °C in accordance with the French standard UEAtc H.1 MOD1, of maximum 500 g/m² and preferably of maximum 180 g/m². The invention also relates to the process for producing such a bonded foam.

WO 01/00718 A1

"A bonded foam and a process for the production thereof."

The present invention relates to a bonded foam comprising a plurality of isocyanate-based polymeric foam particles, in particular polyurethane foam particles, bonded to one another by means of an isocyanate based binder and to a process for the production thereof.

Bonding any kind of polymeric foam is well known as method for recycling and reuse of foams. The density ranges from about 30 kg/m³ to more than 450 kg/m³, where lower densities are suitable for cushioning in the furniture and bedding industry, for mattresses, for applications in the agricultural area and many more. Higher densities (> 250 kg/m³) are being applied preferably for all kinds of floor applications like for instance underlays for parquet, PVC tiles and ceramic tiles.

Bonded foams put on the market before the present invention and having a density range of typically 250 to 400 kg/m³ can absorb up to 250 % at 2 mm thickness (> 1000 g/m² according to UEAtc H.1) of water, depending on the composition, the nature of the foam, the nature and concentration of the binder and the presence of other solid constituents. The lower the density the higher the water absorption under comparable conditions.

Those bonded foams are not suitable for parquet underlays or underlays for PVC- or ceramic tiles under moist conditions like in kitchens or bathrooms or in other applications where floors are cleaned by means of water.

- 2 -

The object of the present invention is therefore to provide a bonded foam which can be used under moist conditions and a process for the production of such a bonded foam.

To this end, the bonded foam according to the invention
5 further comprises at least one water-repellent additive and has a water uptake, determined at 25 °C in accordance with the French standard UEAtc H.1 MOD1, of maximum 500 g/m² and preferably of maximum 180 g/m². The process according to the invention is characterised in that at
10 least one water-repellent additive is incorporated into the bonded foam in such an amount that the bonded foam has a water uptake, determined at 25 °C in accordance with the French standard UEAtc H.1 MOD1, of maximum 500 g/m² and preferably of maximum 180 g/m².

It has been found that, notwithstanding the fact that the foam particles from which the bonded foam is made are usually quite
15 hydrophilic, the water uptake of the bonded foam can be reduced to the indicated values by incorporating a water-repellent additive therein.

In a preferred embodiment, said water-repellent additive is applied substantially superficially onto the polymeric foam particles so that these foam particles are at the most only partially impregnated with
20 the water-repellent additive.

It has indeed been found surprisingly that the polymeric foam particles do not have to be impregnated entirely with the water-repellent additive in order to reduce the water uptake of the bonded foam to the indicated values. In this preferred embodiment, the production
25 costs are considerably reduced. Moreover, it enables the use of solid, in particular powdery or granular water-repellent additives. Even in this latter case, the water uptake could be reduced to the indicated values, notwithstanding the fact that the surfaces of the foam particles, in

- 3 -

particular the inner surfaces thereof, are certainly not entirely coated with the water-repellent additive.

In a further preferred embodiment, said water-repellent additive is selected from the group consisting of fluorochemical dispersions, e.g. fluoroalkyl copolymer mixtures, fluoro aliphatic polymers, fluorocarbon polymers, fluorocarbon emulsions, waxes in particular montan waxes and PE waxes or wax emulsions, ethylene vinylacetate copolymers, PTFE dispersions, silicone products, siloxane emulsions, compounds containing Al-, Zn- and Zr-compounds and combinations of these water-repellent additives, a particular preference being given to a water-repellent additive comprising a fluorochemical dispersion, a wax, and/or a siloxane emulsion.

Other particularities and advantages of the invention will become apparent from the following description of some particular embodiments of the bonded foam and of the process for the production thereof according to the present invention. This description is only given by way of illustrative example and is not intended to limit the scope of protection.

The bonded foam according to the present invention is made from isocyanate-based polymeric foam particles as main starting material. These foam particles are in particular polyurethane (ether or ester), polyurea or polyurea modified polyurethane foam particles. Usually they have a hydrophilic character but in some rare cases they may already contain a water-repellent additive incorporated therein during the foaming process of the prime foam material.

For producing the bonded foam, an isocyanate based binder, having free NCO groups, is applied onto the foam particles. This is usually done by spraying while agitating the foam particles. The foam particles are then preferably vigorously mixed and transferred into a

- 4 -

mould wherein they are compressed and wherein the binder is reacted with an NCO groups reactive compound to bond the foam particles to one another to form the rebonded foam. This can be done by means of steam, superheated steam, hot gases or just by curing at ambient temperature.

The bonded foam is then demoulded, postcured and dried in the usual way. The obtained blocks can be cut into the desired shapes, in particular into blocks, plates, sheets or other geometrical shapes. The bonded foam may be used for different applications, in particular for floor or similar applications. For these latter applications, the foam particles are preferably compressed in such a manner that the produced bonded foam has a density of between 150 and 450 kg/m³ and most preferably a density of between 200 and 450 kg/m³.

In order to modify the properties of the produced bonded foam, use can further be made of solid fillers or other modifiers. These solid fillers may comprise for example rubber or other elastomeric particles such as elastomeric polyurethane particles or possibly chopped textile fibres and/or cork powder as well. They are preferably mixed with the foam particles before applying the binder thereto. The elastomeric particles have preferably a diameter of between 0.05 and 2 mm and most preferably a diameter smaller than 1 mm. The cork powder has preferably a diameter of between 2 and 5 mm and most preferably a particle size of about 3 mm.

The binder used in the process according to the invention may be a binder system formed by a reaction mixture comprising an isocyanate and an active hydrogen containing compound like a polyol. On the other hand, it may also be formed by a prepolymer containing still some free NCO groups which may be cured in particular in the presence of water or water vapour.

- 5 -

The foam particles used in the process according to the present invention are either chopped flocks or cut pieces. Starting from such foam particles, the above described process allows to produce a bonded foam which is especially suited for floor or analogous applications and which has, as already mentioned here above, for example a density of 150 to 450 kg/m³. Due to the hydrophilic character of the used foam particles, the obtained bonded foam has however still much too high water uptake capabilities.

In order to solve this problem, at least one water-repellent additive is incorporated according to the invention into the bonded foam, more particularly in such an amount that the bonded foam has a water uptake, determined at 25 °C in accordance with the French standard UEAtc H.1 MOD1, of maximum 500 g/m² and preferably of maximum 180 g/m².

The water-repellent additive may be incorporated into the bonded foam once it has been demoulded or once it has been cut further into pieces of the desired dimensions. Tests have for example been carried out wherein the bonded foam was drenched in the water-repellent additive and wherein the excess of additive was subsequently squeezed out.

In the method according to the invention preference is however given to incorporating the water-repellent additive into the bonded foam during the production thereof, more particularly before the foam particles are bonded to one another. This can be done in different ways:

- The water-repellent additive may be admixed to the binder before applying this binder to the foam particles;

- 6 -

- In case the water-repellent additive is in liquid form, it can be sprayed onto the foam particles, more particularly before or after applying the binder to these particles and/or even simultaneously thereto; and

5 - In case the water-repellent additive is in solid form, it can be mixed with the foam particles, preferably before having applied the binder thereto so that a homogeneous mixture can more easily be obtained.

10 It should be noted that some water-repellent additives are not liquid as such but can be brought into a liquid form by making an emulsion or a dispersion thereof in a solvent or possibly by dissolving them.

15 In case solid water-repellent additives are applied in the process according to the invention, it is clear that such additives are only applied superficially onto the foam particles so that these particles are not impregnated therewith. It has been found however that the foam particles do not have to be impregnated with the water-repellent additive in order to reduce the water uptake of the bonded foam.

20 Consequently, in a preferred embodiment of the invention, the water-repellent additive is applied also substantially superficially onto the polymeric foam particles in case the additive is in liquid form so that these foam particles are at the most only partially impregnated with the water-repellent additive. This makes the process according to the invention more economically feasible and reduces in particular the production time and costs. Indeed, the water-repellent additive can in this way simply be sprayed onto the foam particles, preferably while
25 these particles are being stirred.

Moreover, the water-repellent additive may thus also be added to the binder notwithstanding the fact that this binder may not entirely impregnate the foam particles since otherwise the foam properties would be lost. In conventional processes for making materials

- 7 -

such as textile materials water-repellent, however, for example in the process disclosed in US-A-3 950 298, the material is completely drenched with an organic solution of the additive so that the water-repellent additive is widely spread onto the inner surface of the textile material and exceeding additive is then squeezed out.

In the process according to the invention, the foam particles have preferably a diameter of between 2 and 20 mm. Most preferably, the foam particles have a diameter of 3 to 5 mm. With these latter sizes of the foam particles, the water-uptake of the bonded foam can be reduced optimally whilst the foam properties are still maintained.

In the process or bonded foam according to the invention, different water-repellent additives can be used. The additive may in particular be selected from the group consisting of fluorochemical dispersions, e.g. fluoroalkyl copolymer mixtures, fluoro aliphatic polymers, fluorocarbon polymers, fluorocarbon emulsions, waxes in particular montan waxes and PE waxes or wax emulsions, ethylene vinylacetate copolymers, PTFE dispersions, silicone products, siloxane emulsions, compounds containing Al-, Zn- and Zr-compounds and combinations of these water-repellent additives.

Experiments have shown that good results can be obtained when the water-repellent additive comprises a fluorochemical dispersion, a wax or wax emulsion, and/or a siloxane emulsion.

When the additive is water-based, one would expect that the quality of the bonded foam would be adversely affected in view of the fact that the NCO-groups of the binder react rather fast with water. However, it has been found quite unexpectedly that the use of a prepolymer instead of a pure isocyanate binder or even an in situ prepolymer (allowing the reaction mixture to react on the foam particles

- 8 -

before spraying the water-repellent additive thereon) enables to obtain a good bonding and a good water-repellency at the same time.

Specific water-repellent additives which can be used according to the invention are :

5 1. Montanwax

A typical example thereof is "Hoechstwax E Pulver Fein" (trademark), a product of Hoechst. This solid product is an ester of montanic acids derived from the oxidative refining of crude montan wax that is extracted from brown coal.

10 These waxes are used as lubricants and internal release agents for the processing of polyvinyl chloride, polyolefins, polyamide, polystyrene, linear polyesters, thermoplastic polyurethanes, etc.

2. Fluorochemical dispersions

15 Several fluorochemicals have been evaluated. The best results were obtained with the fluorochemical "FC-251" of 3M. It is a fluorochemical emulsion that contains 28% of active solids. These active solids are described as a "fluoroalkylcopolymer".

20 The product is used to confer oil and water repellency to fabrics made from natural or synthetic fibers. The emulsion is usually applied by padding on mill pad equipment.

Other trade names of fluorochemical emulsions that have been evaluated and found to be suitable are : FC-3540 (3M), FC-3581 (3M), FC-3551 (3M), FC-3531 (3M), Foraperle 351 (Elf Atochem).

3. Polytetrafluoroethylene

25 An example hereof is "Ceridust 9205 F" produced by the company Hoechst. This ceridust micropowder is a low molecular weight polytetrafluorethylene.

In the known applications the product is incorporated by stirring in paints and coatings, and provides antisticking properties and related surface effects.

4. Siloxane emulsions

5 Tradenames of some typical examples are "BYK LPX 6162" of BYK Chemie and "Tegosivin HE 503" of Goldschmidt. The former is a polyether modified dimethylpolysiloxane, the latter a modified reactive siloxane emulsion water-based.

10 These additives are usually used in the paint industry. They provide more permanent surface properties such as improved solvent and weather resistance, less dirt retention, etc.

5. PE waxes

15 A typical example is "Ceraflour 990" a trademark of BYK Chemie. The product is a micronized polyethylene wax that is used to improve the surface properties of coatings and printing inks.

6. Al-, Zn- and Zr-compounds

20 An example hereof is LEFASOL VH1 of LEFATEX-CHEMIE GmbH. A test was done where a bonded polyurethane foam was impregnated with an aqueous solution of that product. Concentration of active product was 500 g/l H₂O. Excess of liquid was squeezed out. The water uptake of the bonded foam could be reduced in this way until just below 500 g/m².

Examples 1-6

25 In these examples irregular foam particles obtained by chopping a polyether polyurethane foam, having a density of about 30 kg/m³ were used as starting material. The chopped particles had a diameter of about 3 mm. They were mixed with a rubber granulate in a mixer until a homogeneous mix was achieved.

- 10 -

The rubber granulate was comprised of rubber particles having a diameter ranging between 0.1 and 0.6 mm. In case use was made of a solid water-repellent additive, this additive was also mixed first with the foam particles. Then the binder was sprayed onto the solid particles while stirring them. When use was made of a liquid water-repellent additive, this was subsequently sprayed on the solid particles still while stirring them. As binder, use was made of a polyurethane prepolymer binder based on a mixture of 2,4- and 2,6-toluene-diisocyanate and a polyether polyol.

After some additional mixing, the constituents were transferred into a mould (l x w x h = 300 x 300 x 600 mm). The top lid of the mould was closed and the components were compressed to the required density. The top lid was more particularly fixed in a height of 72 mm above the bottom plate of the mould. The reaction of the binder was obtained by pressing superheated steam through the perforated bottom of the mould for about 8 minutes. The bonded foam block was demoulded and allowed to dry and postcure for 24 hours. Out of those blocks suitable samples were cut out, more particularly sheets having a thickness of about 2.5 mm, and the water-uptake was measured by the following method :

Test method UEAtc H.1

This testmethod is used to measure the increase in weight of a sample that is in contact with a humid support.

During the test, a sample is for minimal 60 and maximal 63 minutes positioned on a humid capillary taper , in particular into neutral paper, type "Kleenex". Before starting the test, this capillary taper is completely saturated with a liquid that contains demineralized water to which 1.5 % of a solution consisting of demineralized water and 2 % pure Teepol, a liquid soap, is added. In order to obtain a constant flow of the

liquid, the weight of the taper should be kept constant at 17 g/dm². Dimensions of the taper must be at least 210 x 210 mm. Air bubbles underneath or folds in the capillary taper must be avoided.

5 The taper is positioned on a stiff, flat, non-absorbing support. Dimensions of the support and the sample are identical: 150 x 150 mm. In order to keep the taper sufficiently impregnated, the support is positioned in a bin containing the liquid up to a level which is 3 to 5 mm lower than the upper surface of the support.

10 On top of the sample an upper plate is put. This upper plate must have a weight of 150 g ± 15 g and dimensions of 147 x 147 mm.

The weight of each sample is then determined in dry and wet condition. The increase in weight is calculated and expressed in g/m². For each evaluated formulation, 4 individual samples are tested.
15 The highest uptake in g/m² is reported.

Although the thickness of the sample is not specified in the original test method and although a sample with a higher thickness has a higher initial possibility to absorb a higher amount of liquid, comparative tests have shown that thickness variations, in particular a reduction of
20 the thickness to 1.8 mm, have substantially no influence on the test results when the water-uptake is within the claimed range.

The results of the tests performed in the different examples 1 to 6 are given in the table below.

- 12 -

Ex.	Density	PU foam	Rubber	Prepolymer	Waterrepellent additive		Waterab- sorption
	kg/m ³	%	%	%	%	name	g/m ²
1	300	66	18	16	5	Montanwax (Hoechstwax)	176
2	300	66	18	16	0.5 4.5	FC-251 + montanwax	82
3	300	66	18	16	5	Ceridust 9205 F	484
4	350	66	18	16	5	FC-251	41
5	350	66	18	16	5	TEGOSIVIN HE503	172
6	250	66	18	16	0.5 4.5	BYK LPX 6162 + Ceraflour 990	172

From this table it appears that the water-uptake from the bonded foam can be reduced by means of a solid water-repellent additive, more particularly by powdery montanwax, notwithstanding the fact that such an additive does not penetrate into the foam particles itself (see Example 1). By replacing a small portion of the wax with a fluorochemical emulsion, a significant further reduction of the water-uptake can be obtained (see Example 2). An even larger reduction of the water-uptake can be obtained by using only a fluorochemical emulsion as water-repellent additive. This appears from Example 4, although the water-uptake is also somewhat reduced therein by the higher foam density.

From the above description of some particular embodiments of the bonded foam and the process according to the invention, it will be clear that many modifications can be applied thereto

- 13 -

without departing from the scope of the invention as defined in the appended claims.

In particular, it is possible to add all kinds of further additives to the bonded foam, in particular biocidic agents which protect
5 the bonded foam against biological deteriorations when the bonded foam is subjected to humid conditions.

- 14 -

CLAIMS

1. A bonded foam comprising a plurality of isocyanate-based polymeric foam particles, in particular polyurethane foam particles, bonded to one another by means of an isocyanate based binder,
5 characterised in that the bonded foam further comprises at least one water-repellent additive and has a water uptake, determined at 25 °C in accordance with the French standard UEAtc H.1 MOD1, of maximum 500 g/m² and preferably of maximum 180 g/m².

2. The bonded foam according to claim 1, characterised in
10 that said water-repellent additive is applied substantially superficially onto said foam particles so that these foam particles are at the most only partially impregnated with the water-repellent additive.

3. The bonded foam according to claim 1 or 2, characterised in that said water-repellent additive comprises one or more
15 water-repellent additives selected from the group consisting of fluorochemical dispersions, e.g. fluoroalkyl copolymer mixtures, fluoroaliphatic polymers, fluorocarbon polymers, fluorocarbon emulsions, waxes in particular montan waxes and polyethylene (PE) waxes or wax emulsions, ethylene vinylacetate copolymers, polytetrafluoroethylene
20 (PTFE) dispersions, silicone products, siloxane emulsions, and compounds containing Al-, Zn- and Zr-compounds.

4. The bonded foam according to any one of the claims 1 to 3, characterised in that it further comprises elastomeric particles, in particular rubber and/ or elastomeric polyurethane particles, which
25 particles have preferably a diameter of 0.05 to 2 mm and most preferably a diameter smaller than 1 mm.

5. The bonded foam according to any one of the claims 1 to 4, characterised in that it further comprises a filler material, in particular cork powder and/or textile fibres.

- 15 -

6. The bonded foam according to any one of the claims 1 to 5, characterised in that it contains a biocidic agent.

7. The bonded foam according to any one of the claims 1 to 6, characterised in that it has a density of between 150 and 450 kg/m³ and preferably of between 200 and 450 kg/m³.

8. A process for the production of a bonded foam according to any one of the claims 1 to 7, wherein an isocyanate based binder, having free NCO groups, is applied onto a plurality of isocyanate-based polymeric foam particles, the foam particles having the binder applied thereon are compressed, and the binder is reacted with an NCO groups reactive compound to bond the foam particles to one another, characterised in that at least one water-repellent additive is incorporated into the bonded foam in such an amount that the bonded foam has a water uptake, determined at 25 °C in accordance with the French standard UEAtc H.1 MOD1, of maximum 500 g/m² and preferably of maximum 180 g/m².

9. The process according to claim 8, characterised in that said water-repellent additive is applied substantially superficially onto the polymeric foam particles so that these foam particles are at the most only partially impregnated with the water-repellent additive.

10. The process according to claim 8 or 9, characterised in that said water-repellent additive is admixed to the binder before applying this binder to the foam particles.

11. The process according to any one of the claims 8 to 10, characterised in that said water-repellent additive is sprayed in liquid form onto the foam particles before, after and/or simultaneously to applying the binder thereto.

12. The process according to any one of the claims 8 to 11, characterised in that said water-repellent additive is mixed in solid form

- 16 -

with the foam particles, preferably before having applied the binder thereto.

13. The process according to any one of the claims 8 to 12, characterised in that said water-repellent additive is selected from the group consisting of fluorochemical dispersions, e.g. fluoroalkyl copolymer mixtures, fluoro aliphatic polymers, fluorocarbon polymers, fluorocarbon emulsions, waxes in particular montan waxes and polyethylene (PE) waxes or wax emulsions, ethylene vinylacetate copolymers, polytetrafluoroethylene (PTFE) dispersions, silicone products, siloxane emulsions, compounds containing Al-, Zn- and Zr-compounds and combinations of these water-repellent additives.

14. The process according to claim 13, characterised in that said water-repellent additive comprises a fluorochemical dispersion, a wax or wax emulsion, and/or a siloxane emulsion.

15. The process according to any one of the claims 8 to 14, characterised in that use is made of foam particles having a diameter of 2 to 20 mm and preferably of 3 to 5 mm.

16. The process according to any one of the claims 8 to 15, characterised in that at least one solid material, in particular a filler material such as cork powder and/or textile fibres and/or an elastomeric material such as rubber and/or elastomeric polyurethane particles, are incorporated in the bonded foam by mixing them with said foam particles, preferably before applying the binder.

17. The process according to any one of the claims 8 to 16, characterised in that use is made of foam particles having already a water-repellent additive incorporated therein, which water-repellent additive is either different from or the same as the water-repellent additive which is incorporated in the bonded foam.

- 17 -

18. The process according to any one of the claims 8 to 17, characterised in that the isocyanate-based polymeric foam particles are compressed in such a manner that the produced bonded foam has a density of between 150 and 450 kg/m³ and preferably of between 200 and 450 kg/m³.
- 5

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08J9/33 //C08L75/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 43 13 781 C (DAIMLER BENZ AG ;METZELER SCHAUM GMBH (DE)) 4 August 1994 (1994-08-04)	1-3, 8, 9, 13, 15
Y	page 2, line 37-60 example 1 claims	5
Y	FR 2 097 144 A (GEN FOAM PRODUCT LTD) 3 March 1972 (1972-03-03) claims	5

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents:

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INTERNATIONAL SEARCH REPORT

Information on patent family members

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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